AFRL-SR-BL-TR-00-

0021

REPORT DOCUMENTATION PAGE

1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
12 October, 2000	Technical report	3/1/98-2/28/99

4. TITLE AND SUBTITLE
Integrated Instrumentation for Light-Emitting Polymers Development

5a. CONTRACT NUMBER

5b. GRANT NUMBER
F49620-98-1-0327

5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

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10. SPONSOR/MONITOR'S

20001120 195

12. DISTRIBUTION / AVAILABILITY STATEMENT

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED

13. SUPPLEMENTARY NOTES

14. ABSTRACT

The major objective of this project is to develop an integrated instrumentation that combines the capability of performing spin-coating of uniform polymer thin films under an oxygen and moisture free environment, metal evaporation and organic molecular deposition with controllable thickness. In addition, it provides timely assessment of electroluminescent material properties through the measurements of thermal properties (Tg and decomposition temperature), conductivity, re-dox potentials, as well as the photo- and electro-luminescence emission spectra. By integrating these testing functions together, it provides a very efficient mechanism to evaluate potential polymer systems for the fabrication of light-emitting devices. This integrated instrumentation is available for scientists in the research community (both from academic and DoD laboratories) to perform a reliable screening and testing procedure and to speed up the development of LED polymer material systems. This, in turn, will provide the technological basis for the design and fabrication of reliable polymer LEDs.

15. SUBJECT TERMS

POLYMERS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Alex K-Y. Jen
a. REPORT	b. ABSTRACT	c. THIS PAGE			19b. TELEPHONE NUMBER (Include area code)
Unclas	Unclas	Unclas		7	(206) 543-2626

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

END-OF-THE-YEAR-REPORT

for

GRANT #, F49620-98-1-0327

PR Number, F08671-9801136

Integrated Instrumentation for Light-Emitting Polymer Development

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October 1, 2000

1. Introduction

There is a growing interest in ultilizing polymeric light-emitting diodes (LEDs) in large area flat panel displays. They possess significant processing, mechanical, thermal, and size advantages over those made by evaporation methods. In addition, it is possible to fabricate full color displays utilizing the broad spectral range of emission colors available from semiconducting luminescent polymers and organic molecules. The potential for low-cost manufacturing, packaging, and assembly arises from the demonstrated ability to perform multi-layer integration of the polymers and their excellent compatibility with integrated circuits. In principle, polymer LEDs require the injection of holes and electrons into the emitter layer. The recombination of the injected electrons and holes in the polymer layer generates singlet excitons whose radiative decay produces visible light. The characteristics of a polymer LED are determined by the tunneling of both holes and electrons through the interface barriers which is caused by the band offset between the electroluminescent polymer and the electrodes. A significant difference in the barrier height at the polymer/cathode and polymer/anode interfaces results in unbalanced hole and electron injections and therefore dramatically reducing the photon/electron quantum efficiency of the devices. In order to achieve higher device efficiency, highly luminescent polymers must be chosen, and balanced charge (hole and electron) injection is considered to be crucial.

The greatest challenge for scientists in this field is to find suitable material systems (light emitter, electron and hole transporters) which possess efficient and balanced injection of holes and electrons to ensure low operating voltage and high quantum efficiency, with matched redox potentials for metal electrode selections, and good mechanical properties for multi-layer integration. In addition, it is essential to improve the processing and packaging techniques in order to improve the operating lifetime of these devices. Although different groups are developing the basic luminescent molecules, polymeric materials, processes, and devices, they measure and report their results based on different specific and individual tests. Due to the different test procedures and measurement methods, it is difficult to make comparisons between the materials. Thus, selecting the most promising development path becomes difficult. To speed up the tedious selection process, it is highly desirable to have an integrated instrumentation that provides the necessary information such as spin-coating and metal evaporation protocols, redox

potentials, photo- and electroluminescence emissions, current-electric field characteristics, and thermal properties of the polymers, in a short time span.

2. Objective

The major objective of this project is to develop an integrated instrumentation that combines the capability of performing spin-coating of uniform polymer thin films under an oxygen and moisture free environment, metal evaporation and organic molecular deposition with controllable thickness. In addition, it provides timely assessment of electroluminescent material properties through the measurements of thermal properties (Tg and decomposition temperature), conductivity, redox potentials, as well as the photo- and electro-luminescence emission spectra. By integrating these testing functions together, it provides a very efficient mechanism to evaluate potential polymer systems for the fabrication of light-emitting devices. This integrated instrumentation is available for scientists in the research community (both from academic and DoD laboratories) to perform a reliable screening and testing procedure and to speed up the development of LED polymer material systems. This, in turn, will provide the technological basis for the design and fabrication of reliable polymer LEDs.

3. Impact to the new research program on LED materials at the Northeastern University

The integrated instrumentation has greatly enhanced the quality and capability of the new LED materials research at Northeastern University (NU) to evaluate suitable LED material system properties. The new facility established by Professor Alex Jen possesses the capability of synthesizing and characterizing novel conjugated polymers. This integrated instrumentation has helped to guide the synthetic effort to fine-tune the properties of molecular structures and establish desirable LED polymeric material system properties, and thus, directly impact the fabrication of highly efficient EL devices. Seventeen light-emitting polymers related papers have been published in the refereed journals based on the characterization results derived from this set-up. In addition, this facility has provided very useful services to researcher (Professor Timothy Swager-MIT, Professor Lin Pu-U. of Virginia, Professor Larry Dalton-U. of Washington) that are supported by DoD's funding agency. The capability of this integrated instrumentation includes the spin-coating of uniform polymer thin films in a dry box, metal evaporation and organic molecuar deposition with controllable thickness, rapid assessment of

material properties by the measurements of thermal properties (Tg and decomposition temperature), conductivity, redox potentials, and photo- and electro-luminescence emission spectra of the polymers .

4. Interface between the instrumentation and the existing facility for electro-optic (E-O) materials research at the Northeastern University

This integrated instrumentation interfaces very well with the existing E-O materials research facility at Northeastern University (NU) to jointly evaluate organic photonic/opto-electronic material properties. One of the new research program proposed by both professors Alex Jen and Yang Yang (UCLA) aims at demonstrating an integrated all polymer LED/E-O device by using organic conjugated polymers as both a light source (LED) and a photodetector, and using NLO polymer channel waveguides as an E-O switching device. This instrumentation greatly enhances the capability of quickly developing/screening both LED and E-O materials systems to ensure the greatest chance of success. In the area of polymer characterization, the facility at NU is equipped with the instruments such as TGA and DSC for thermal analysis; GPC and HPLC for polymer molecular weight measurement; and Dektak instrument for measuring thin film thickness. In addition, FT-IR and UV-Vis-Near IR spectrometer were used to detemine the thermal stability of the E-O polymer thin films. In the areas of optical and electrial characterization, the micromanupilator device could be used to cure (up to 400 °C) and pole NLO thin films and channel waveguides; Metricon prism coupler could measure refractive index, optical loss, and thickness of polymer thin films; lock-in amplifier and the associated electronic system could measure optical and electro-optic signal generated by LED/E-O materials. This integrated instrumentation will help to bridge between the effort of evaluating E-O and LED polymeric material system properties, and thus, directly impact the fabrication of all polymer LED/E-O devices.

5. Research training of students

The highly interdisciplinary nature of the program to develop high performance LED materials for device applications, the outstanding faculty and institutions involved, and connections with high technology device companies and DoD laboratories ensure a rich educational environment for the graduate students, postdoctors, and undergraduate students involved. Students are active members involved in closely integrated material synthesis, characterization, and device fabrication. Students associated with this program will emerge with a unique background and complement of skills. The ability to communicate with and work with academic, government, and industrial researchers in other disciplines towards a common goal will uniquely qualify them for the technical workforce of the future.

6. Papers published that acknowledge the AFOSR

1. "Design and Synthesis of Luminescent Polymers with both Electron Withdrawing and Electron Donating Groups", X. C. Li, A. K-Y. Jen, Y. Q. Liu and S. Liu, Polymer Preprint, 1998, **39(2)**, 1093.

2. "Synthesis and Characterization of Quinoline-Triphenyldiamine Copolymers as Light-emitting Materials", Y. Q. Liu, H. Ma, S. Liu, X-C. Li, and A. K-Y. Jen, Polymer Preprint, 1998, **39(2)**, 1089.

3. "Synthesis and Characterization of a Novel and Highly Efficient Light-emitting Polymer", Y. Liu, M. S. Liu, and A. K-Y. Jen, Acta Polymerica, 1999, **50**, 105.

4. "Synthesis and Characterization of a Novel Light-emitting Polymer Containing Highly Efficient Hole-transporting Aromatic Diamine", Y-Q. Liu, M. S. Liu, X-C. Li, and A. K-Y. Jen, Chem. Mater., 1998, **10(11)**, 3301.

5. "Synthesis and Characterization of a Bipolar Light-emitting Copolymer Consisting of Tetraphenyldiaminobiphenyl and Bis-quinoline Units", Y-Q. Liu, H. Ma, and A. K-Y. Jen, Chem. Mater., 1999, **11 (1)**, 27.

6. "Synthesis and Characterization of a Novel Bipolar Polymer for Light-emitting Diodes", Y-Q. Liu, H. Ma, and A. K-Y. Jen, Chem. Commun., 1998, 2747.

7. "Synthesis, Properties and Applications of New Luminescent Polymers with Both Hole and Electron Injection Abilities for Light-emitting Devices", X-C. Li, Y. Liu, M. S. Liu, and A. K-Y. Jen, Chem. Mater.,1999, **11**, 1568.

8. "Synthesis and Characterization of Polyquinolines for Light-emitting Diodes", M. S. Liu, Y. Liu, C. Urian, H. Ma and A. K-Y. Jen, J. Mater. Chem., 1999, **9(9)**, 2201.

9. "Synthesis and Characterization of a High Performance Co-polymer for Light-emitting Diode", Y-Q. Liu, H. Ma, M. S. Liu, S. Liu and A. K-Y. Jen, Proc.SPIE , 1999, **3623**, 28.

10. "Polyquinolines: Multifunctional Polymers for Electro-optic and Light-emitting Applications", A. K-Y. Jen and H. Ma, Mat. Res. Soc. Proc. (in press).

11. "Efficient Light-emitting Diodes Based on a Novel Binaphthalene-containing Polymer", A. K-Y. Jen, Y. Liu,Q. Hu and L. Pu, Appl. Phys. Lett. 1999, 75(24), 3745."A Binaphthyl-Based Conjugated Polymer for Light-Emitting Diodes", L. Zheng, R. C. Urian, Y. Q. Liu, A. K-Y. Jen and L. Pu, Chem. Mater., 2000, **12(1)**, 13.

12. "High Performance Binaphthyl-Based Polymers for Light-Emitting Devices", L. Zheng, X. Jiang, S. Liu, and A. K-Y. Jen, J. Organometallic Chem. (in press).

13. "High Performance Blue Light-Emitting Diode Based on a Binaphthyl-Containing Polyfluorene", X. Jiang, S. Liu, H. Ma, and A. K-Y. Jen, Appl. Phys. Lett. 2000, 76(14), 1813.

14. "Tuning of Redox Behavior and Fluorescence of Cyano-containing Phenylenevinylene Oligomers and Polymers", M. S. Liu, X. Jiang, and A. K-Y. Jen, Mat. Res. Soc. Proc. 1999, xxx. (in press)

15. "1,1-Binaphthyl-Containing Polyfluorenes for Efficient Light-Emitting Diodes", S. Liu, X. Jiang, M. S. Liu, H. Ma, and A. K-Y. Jen, Mat. Res. Soc. Proc., 1999, xxxx

16. "Polybinaphthylenevinylene-alt-phenylene vinylene) Derivatives: Novel Luminescent Polymers for Light-Emitting Devices", L. Zheng, X. Jiang, M. S. Liu, and A. K-Y. Jen, Mat. Res. Soc. Proc., 1999, xxx.

17. "Organic Light-Emitting Diodes Using a in-situ thermally polymerized hole transporting layer", X. Jiang, S. Liu, M. S. Liu, H. Ma, and A. K-Y. Jen, Appl. Phys. Lett. 2000, **76(21)**, 2985.

18. "Triarylamine-Containing Poly(perfluorocyclobutane)s as Hole-Transporting Materials for Polymer Light-Emitting Diodes", S. Liu, X. Jiang, H. Ma, and A. K-Y. Jen, Macromolecules, 2000, **33**, 3514..

19. "Efficient Light-Emitting Devices with Polyfluorene Emitting Layer Thermally Polymerized Amine-Containing Hole-Transporting Layer", X. Jiang, S. Liu, H. Ma, L. Zheng, M. Liu, and A. K-Y. Jen, Poly. Mater. Sci. Eng., 2000, **83**, 204.

20. "Organic Electroluminescent Devices Based on Phenanthrene-Containing Eurropium Complex", D. Huang, X. Jiang, G. Phelan, T. Londergan, A. K-Y. Jen, and L. R. Dalton, Poly. Mater. Sci. Eng., 2000, **83**, 266.

List of the equipment purchased

Equipment List

EQUIPMENT	MODEL	UNIT PRICE	TOTALS	VENDER AND ADDRESS
HP Semiconductor Parameter Analyzer	4155A	$ 33,300		Hewlett Packard
English Manual Set	Option ABA			P. O. Box 4026
For 50 Hz Line Frequency	Option 050			Englewood, CO 80155-4026
for 60 Hz Line Frequency	Option 060			Tel:1-800-829-4444
				Fax: 1-800-829-4433
subtotal			$ 33,300	

Photodiode Detector Dual Channel High Performance Optical Meter	818-UV/an 2835 C	$ 800 $ 5200		Newport Corporation Attn:Order Entry Department P.P Box 19607 Irvine , CA 92713-9607 Tel:1-800-222-6440 fax:1-714-253-1680
subtotal			$ 6,000	
Dri-Lab, one work station Mini-Antechamber Butyl Rubber Gloves Glove Port Cover Chemical Rack and Shelf Assembley Fluorescent Skylight Filter Assembly For Aluminum Labs Pump, Vacuum	HE-43-2 AC-4A 8B3032L&R HE-213-4 DL-CHEM RACK HE-303 FLTR-04 VP-02	$ 26,000		Vacuum Atmospheres Company 4652 West RoseCrans Avenue PO Box 1043 Hawthorne, CA 90250 Tel: 1-310-644-0255 Fax: 1-310-970-0980
subtotal			$ 26,000	
Custom Vacuum Evaporator with two rate/thickness monitors & conical tops and shutters	CV301M	$ 30,000		Cooke Vacuum Products 13 Merritt Place South Norwalk, CT 06854 Tel:1-203-853-9500 Fax: 1-203-838-9553
subtotal			$ 30,000	
Photo Resist Spinner chuck (3/4") chuck (3-5/16")	EC101DT-R790 2-02704 2-02712	$ 6,375 $ 221 $ 389		Headway Research , Inc. 3713 Forest Lane Garland, Texas 75042-6928 Tel:1-972-5431 Fax:1-972-272-7817
subtotal			$ 6,985	
Integrating Sphere (sphere) Sample Holder Port Aperture Port Aperture Port Converter Port Converter Light Trap Dector Adaptor Part frame light Trap	819-IS-4 819-SH 819-PA-0.5 819-PA-1.0 819-PC-0.5 819-PC-1.5 819-LTSH 819-DA 819-LTP	$ 2,137 $ 283 $ 295 $ 75 $ 155 $ 211 $ 80 $ 211		Newport Corporation Attn: Order Entry Department P.O. Box 19607 Irvine , CA 92713-9607 Tel:1-800-222-6440 Fax:1-714-253-1680
subtotal			$ 3,447	
InstaSpec IV CCD Detection System MS125 1/8th Meter Focal Length Spectrograph Mounting Flange for MS125 Grating:400 1/mm blazed at 500 nm Fiber Optic Mounting Kit Single Core Fiber-400nm diameter 100 um slit SMA to 11 mm diameter ferrule	78401 77400 77439 77417 77860 77511 77223 77670	$ 14,175 $ 1,687 $ 139 $ 273 $ 101 $ 292 $ 126 $ 32		Oriel Instruments 250 Long Beach Blvd. Stratford, CT 06497-0872 Tel: 1-203-377-8282 Fax: 1-203-378-2457
subtotal			$ 16,825	
TOTAL			$122,557	